Technical Report No. 32-36

Time and Temperature Dependence of the Ultimate Properties of an SBR Rubber at Constant Elongations

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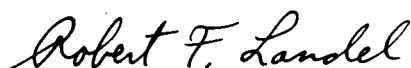
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ABSTRACT

The ultimate properties of amorphous rubbers at temperatures above T_g are considered in terms of stress-strain curves to rupture measured at different strain rates and temperatures. The consideration indicates that a specimen held at a fixed elongation should break eventually, provided the elongation exceeds a critical value. This expected behavior was found by studying an SBR rubber. For samples maintained at different elongations for up to seven days, both the time-to-break and the stress-at-break were measured at eight temperatures from 1.7 to 60°C. For comparison, the ultimate properties were also measured at different strain rates and temperatures. The comparison indicates that for a given ultimate elongation and stress-at-break, the time-to-break under conditions of constant elongation is less than under conditions of constant strain rate.

I. INTRODUCTION

A study was made previously of the temperature and strain rate dependence of the stress-at-break (tensile strength) and the ultimate elongation of an unfilled SBR rubber (Ref. 1). In this study, stress-strain curves to the point of rupture were measured with an Instron tensile tester on ring-type specimens at fourteen temperatures between −67.8 and 93.3°C and at eleven strain rates between 0.158×10^{-3} and 0.158 sec^{-1} at most temperatures. The tensile strength was found to increase with both increasing strain rate and decreasing temperature. At all temperatures above −34.4°C, the ultimate elongation was likewise found to increase with increasing strain rate and decreasing temperature, but at lower temperatures the opposite dependence on rate was observed; at −34.4°C, the ultimate elongation passed through a maximum with increasing rate.

Values of the ultimate properties at each temperature were plotted against log $1/R$, where R is the strain rate. The resulting curves were then superposed by shifting them along the log $1/R$ axis, the shift distance required to effect superposition being log a_T. Values of log a_T obtained from superposing the tensile strength data were equal, within experimental error, to those obtained by superposing the ultimate elongation data. Further, these values of log a_T were found to fit the Williams, Landel, and Ferry (WLF) equation (Ref. 2)

$$\log a_T = -\frac{8.86\,(T - T_s)}{101.6 + T - T_s} \tag{1}$$

with the standard reference temperature T_s equal to 263°K. In this form of the WLF equation, T_s is normally about 50° higher than the glass temperature T_g of a

material. Thus, the value obtained for T_s indicated T_g should be 213°K, in close agreement with 210°K obtained from dilatometric measurements. Values of a_T given by Eq. (1) were used to construct two reduced curves which showed all ultimate property data plotted against log $1/Ra_T$. These curves showed the strain-rate dependence of the tensile strength and the ultimate elongation, over many decades, at the standard reference temperature.

In studying the linear viscoelastic properties of amorphous polymers above T_g, many workers have found the shift factor a_T to be extremely useful for interconverting the effects of temperature and experimental time-scale. From the study of the SBR rubber, it was concluded that a_T can also be used to interconvert the effects of temperature and strain rate on the ultimate properties of certain types of polymeric materials. Prior to this work, F. Bueche (Ref. 3) presented a theory which predicted that values of the tensile strength measured at different temperatures and under either constant loads or strain rates can be interrelated by using a_T values. To check the theory, Bueche measured the tensile strength of polybutyl methacrylate under constant loads at temperatures above T_g and applied time-temperature superposition to reduce the data to a single temperature.

The shift factor is given by the relation $a_T = (T_0/T)$ (η/η_0), where η is the viscosity (or internal viscosity for crosslinked polymers) at temperature T, and η_0 is the viscosity at some arbitrary reference temperature T_0. Because T_0/T differs from unity by a relatively small amount, a_T equals approximately the viscosity ratio η/η_0, and consequently the temperature dependence of a_T is essentially the same as that of the viscosity. Thus, the observation that a_T can be used to interconvert the effects of strain rate and temperature on the ultimate properties —at least for materials like SBR rubber—leads to the following conclusions: (1) the ultimate properties vary with temperature because the internal viscosity varies with temperature; (2) the ultimate properties vary with strain rate because the viscous resistance to network deformations increases with the rate; and (3) the temperature dependence of factors such as the rate of bond rupture have no significant effect on the temperature dependence of the ultimate properties.

In Fig. 1 values of the ultimate properties shown by the reduced curves in the previous paper (Ref. 1) have now been replotted against the time-to-break (t_b) expressed in seconds and reduced to 263°K; t_b equals γ_b/Ra_T, where γ_b is the strain at break measured at a strain rate R and temperature T, and S_b is the stress-at-break based on the initial cross-sectional area of the
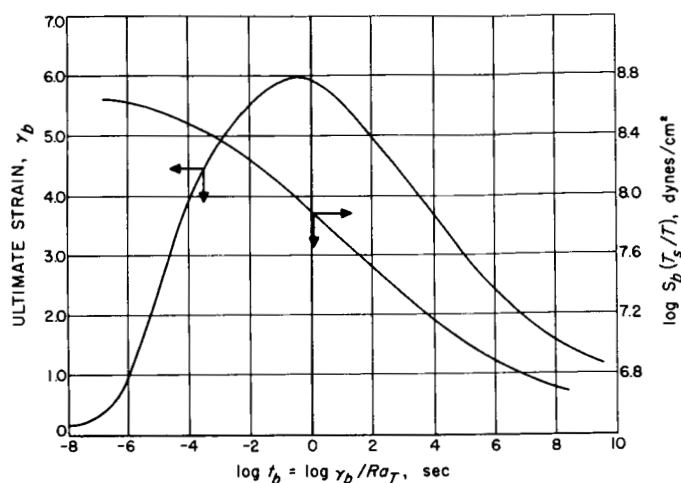
Figure 1. Ultimate properties of an SBR rubber measured at different strain rates and temperatures

Data plotted against the logarithm of the time-to-break (t_b) reduced to −10°C. (Data from Ref. 1.)

specimen. Figure 1 shows that the ultimate properties become relatively insensitive to strain rate at sufficiently low rates; the shapes of the curves suggest that asymptotes may exist which have near-zero slopes. Because of kinetic effects which must be associated with the chain scissions which lead to specimen rupture, it is unlikely that asymptotes of zero slope exist. However, the asymptotic or long-time values of the ultimate properties are of major significance because such values are nearly independent of the testing conditions and are characteristic of the network in the absence of viscous effects.

An important qualitative conclusion about the time-dependent ultimate properties can be reached by examining Fig. 1. For example, this Figure shows that a specimen will break at 300% elongation provided the strain rate is so selected that 10^5 sec are required to reach this elongation. Let us suppose, however, that a specimen is stretched at the strain rate needed to produce a 300% elongation in 100 sec and that the elongation is subsequently held constant. Immediately after the sample is stretched, the stress will begin to relax because the sample was not stretched under equilibrium conditions. Although the stress decays, the sample will break eventually because it can withstand an elongation of only about 100% when the network chains are in an equilibrium condition. As a rough approximation, it might be expected that the sample will break after a total of 10^5 sec, as indicated by the curve which shows the ultimate strain plotted against log t_b. However, this curve represents data measured under conditions of constant strain rate, and it is to be expected that values of the ultimate properties,

like other time-dependent mechanical properties, will depend on the path.

Important conclusions about the time dependence of the ultimate properties can also be reached by considering the diagram in Fig. 2, which shows stress-strain curves, including the rupture points, measured at various strain rates and temperatures. The variation of the stress-at-break and the strain-at-break with either strain rate or temperature is shown by the envelope ABC. The curve OA represents equilibrium stress-strain conditions, except possibly in the vicinity of point A. Although curve OA represents stress-strain values in the absence of viscous effects, true equilibrium— especially in the vicinity of A —undoubtedly does not exist because under suitable conditions stress leads to the breakage of chemical bonds. Thus, values of the ultimate properties are not expected to be unique but to depend on the temperature and stress history. However, for present purposes, we can ignore such degradation effects and assume that A represents the ultimate properties measured under equilibrium conditions.
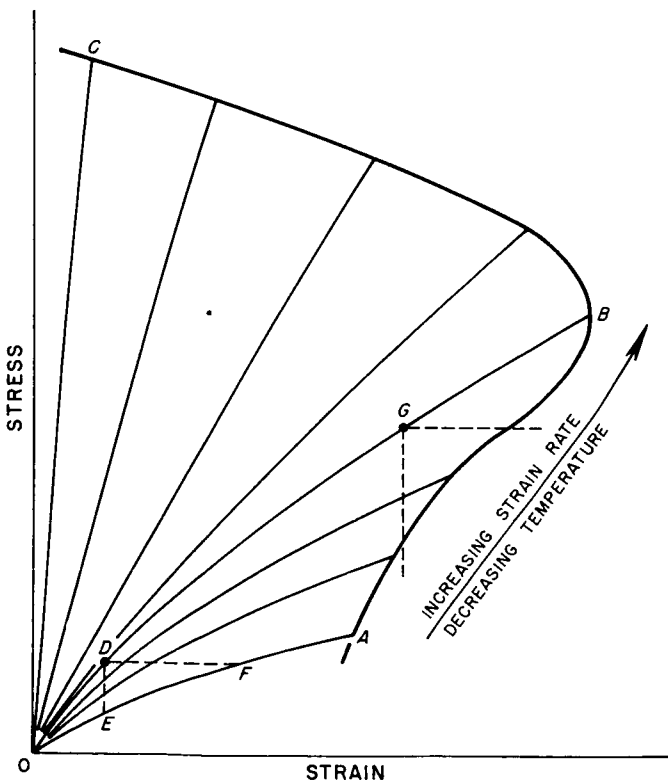
Figure 2. Schematic representation of the variation of stress-strain curves with strain rate and temperature

Envelope connects rupture points and the dotted lines illustrate stress relaxation and creep under different conditions

Suppose now that a specimen is stretched at the strain rate and temperature required to traverse curve OB and that the strain is held constant after point D is reached. At the constant strain, the stress relaxes, as indicated by the dotted vertical line, until it reaches E, which is on the equilibrium stress-strain curve. On the other hand, if the stress is held constant after point D is reached, the strain increases, as indicated by the dotted horizontal line, until it reaches point F, which is also on the equilibrium stress-strain curve.

Next suppose that curve OB is traversed until point G is reached, and thereafter the strain is held constant. The stress again relaxes, but because no equilibrium stress exists for the strain represented by point G, the specimen should eventually break. Likewise, if the stress is held constant after point G is reached, the strain increases, and the specimen should eventually break because no equilibrium strain exists for the stress represented by point G.

Studies have been made of the time-to-break for various materials under constant loads (see, for example, Refs. 4 and 5), although with several possible exceptions (Refs. 3 and 6), most studies have been made of rigid materials. Apparently no studies have been made of ultimate properties under constant strain conditions. Thus, the primary purpose of the present work was to obtain experimental confirmation of the prediction that certain rubbers, when held at constant elongation, will rupture eventually, provided the elongation exceeds some critical value; the secondary purpose was to see how values for the ultimate elongation and stress-at-break[1] measured under constant strain conditions compare with values measured under constant strain-rate conditions. To obtain the desired data, measurements were made at various temperatures of the stress-relaxation of an SBR rubber under relatively large elongations; the apparatus used and the stress-relaxation data obtained are discussed in detail elsewhere (Ref. 7). This Report presents data on the time required for the rupture of specimens subjected to various elongations at various temperatures and the stress existing at the time of rupture. For comparative purposes, a limited study was also made of the ultimate properties of the SBR rubber at different strain rates and temperatures.

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[1]Because tensile strength is usually defined as the maximum stress developed during certain types of tests, it seems desirable here to speak of the stress-at-break and to call the stress-at-break the tensile strength only when it equals the maximum stress developed during a test.

II. EXPERIMENTAL

A. Material

Ultimate properties were measured of a gum vulcanizate of a styrene-butadiene rubber (SBR) which was supplied in the form of ASTM tensile sheets (ca. 0.080 in. thick) by the Rubber Section of the National Bureau of Standards. The base polymer was NBS Reference Sample 387 (X-768) which contained 23.5% bound styrene and had been prepared by emulsion polymerization at 50°C. The gum vulcanizate, which was cured for 60 min at 150°C, contained the following parts by weight: SBR, 100 parts; zinc oxide, 5 parts; sulfur, 2 parts; and benzothiazyl disulfide, 1.75 parts. The sheets of the vulcanizate were stored in a freezer prior to being used. The vulcanizate used in the previous study (Ref. 1) was the same except that 1 part of mercaptobenzothiazole was used in place of 1.75 parts of benzothiazyl disulfide and cure was for 45 instead of 60 min.

The average molecular weight between crosslinks M_c for the vulcanizate was determined from swelling measurements in four solvents. Duplicate weighed pieces of SBR were immersed in the solvents for four days at 25°C, and the swollen samples were weighed after removing excess solvent from their surfaces with filter paper. Values of M_c were calculated using the equation

$$\ln(1 - v_2) + v_2 + \chi_1 v_2^2 = - \rho \frac{V_1}{M_c}\left(v_2^{1/3} - \frac{v_2}{2} \right)$$

where v_2 is the volume fraction of polymer in the swollen gel, ρ is the density of SBR, V_1 the molar volume of the solvent, and χ_1 the solvent-polymer interaction parameter. Values of χ_1, approximated as described previously (Ref. 1), are given in Table 1 along with the values of M_c for both the present vulcanizate and the vulcanizate used in the previous study.

A value of M_c was also calculated from the tensile modulus E derived from stress-strain data measured at 80°C with the Instron tensile tester. The modulus was

evaluated, as described elsewhere (Ref. 8), from the slope of a plot of αS vs γ, where αS is the stress based on the stressed cross-sectional area of a specimen, and γ is the strain. At 80°C, the modulus was found to decrease from 7.35 to 6.58 × 10⁶ dynes/cm² as the strain rate of the test was decreased from 1.516×10^{-2} to 1.516×10^{-4} sec⁻¹. At 60°C and a strain rate of 1.516×10^{-3}, the modulus was 6.96×10^6 dynes/cm². These data indicate that the value measured at the lowest rate at 80°C is a near-equilibrium one. By substituting $E = 6.58 \times 10^6$ at $T = 353°K$ in the equation $M_c = 3 \rho RT/E$, it is found that $M_c = 13,100$, in fair agreement with 15,900 obtained from swelling data (Table 1).

Unfortunately, M_c values for the vulcanizate studied previously were 8020 and 9250, as obtained from swelling and modulus data, respectively. The difference in M_c of the two vulcanizates precludes quantitative comparisons of the ultimate properties of the first vulcanizate, studied at constant strain rates, with those observed on the present vulcanizate under constant strain conditions. Because of the desire to compare results obtained under these different conditions, some measurements under constant strain rates were also made on the present vulcanizate.

B. Apparatus

Data under both constant strain-rate and constant strain conditions were measured on rings which were cut from the sheets of SBR with a circular die which had an inside diameter of 1.400 in. and an outside diameter of 1.650 in. The edges of the die-cut rings were dished slightly, although this slight imperfection was disregarded in measuring the dimensions of specimens. Tensile data were measured at different temperatures and strain rates with an Instron tensile tester equipped as briefly described previously (Ref. 1).

The constant strain apparatus (Ref. 7) contained eight racks for stretching ring specimens. These racks were mounted in an air thermostat whose temperature was controlled by the continuous circulation of heated or refrigerated air from an external source. Temperature control devices maintained the temperature at any given point to within 0.5°C for many hours, and the maximum temperature difference between various points in the thermostat was about 1°C. When the temperature was regulated below ambient for several days or more, the over-all temperature variation, as indicated by three

Table 1. Values of M_c from equilibrium swelling data

Solvent	χ_1	Previous vulcanizate	Present vulcanizate
Carbon tetrachloride	0.300	8000	16,000
Xylene	0.305	8700	16,600
Toluene	0.316	8000	16,000
Benzene	0.345	7400	14,800
Av.		8000	15,900

thermocouples, was about ±1.5°C, this large variation being due to certain operational changes produced by frosting of the refrigeration coils. During operation, a stream of dry nitrogen was bled continuously into the system to delay frosting.

Each rack for stretching a specimen consisted of a vertically mounted brass rod on which two movable supports were attached. Each support had a freely rotating nylon pulley whose diameter was 0.150 in., and a ring-type specimen was placed over the pulleys. The upper support was connected to a heat-treated steel beam which rested on knife-edges mounted on top of the thermostat, and the lower support was connected by a cable to a wheel mounted under the thermostat. By adjusting the position of a metal stop on the brass rod, the specimen could be stretched manually by the wheel and cable device to a predetermined elongation in 2 sec or less. The force exerted by a stretched specimen was determined by measuring the small deflection of the beam with a linear variable differential transformer whose output was recorded by a Brown potentiometer; the output was determined to be a linear function of the applied force within about 1%.

Ultimate properties under constant strain conditions were measured on eight samples simultaneously. To carry out tests, unstrained rings of SBR were placed over the pulleys on the racks and allowed to come to temperature equilibrium. The rings were then rapidly stretched to predetermined elongations, the elongation increment of adjacent specimens normally being 25%. The elongations were calculated from the final distance between the centers of the pulleys, the diameter of the pulleys, and the initial inside diameter of a ring. The inside diameter was used to obtain the maximum elongation existing in a stretched ring.

Other experimental work has indicated that the maximum elongation, instead of some average value, correlates best with the ultimate elongation measured on tensile specimens in which the strain is uniform. To obtain the stress, the measured load on the supporting beam was divided by twice the initial cross-sectional area of the ring. Because of some uncertainties in determining the absolute value of the load during a measurement, and in determining the cross-sectional area of a specimen, the error in the stress may at times have been as great as 10%.

III. RESULTS AND DISCUSSION

Stress relaxation measurements were made at various elongations at eight temperatures between 1.7 and 60°C, most measurements being continued until rupture occurred. (A few measurements were also made at −5 and −15°C, but the rupture data were erratic and thus are not presented.) Stress relaxation data obtained during a typical run are shown in Fig. 3. These data were measured at 1.7°C and at eight elongations between 350 and 525%. The solid points indicate when rupture occurred, and the dotted curve through these points shows the stress-at-break at the various elongations.

Plots are shown in Fig. 4 of the percentage of elongation against the logarithm of the time-to-break (t_b) for results obtained at 1.7, 7.2, 12.8, and 18.3°C; data obtained at 29.4, 40, and 50°C are shown in Fig. 5. Two sets of measurements were made at each temperature and the results from each are differentiated by flags on the points. Figures 6 and 7 show plots of log S_b 286/T vs log t_b, where S_b is the stress-at-break and T is the absolute temperature of measurement. As shown in Fig. 6,
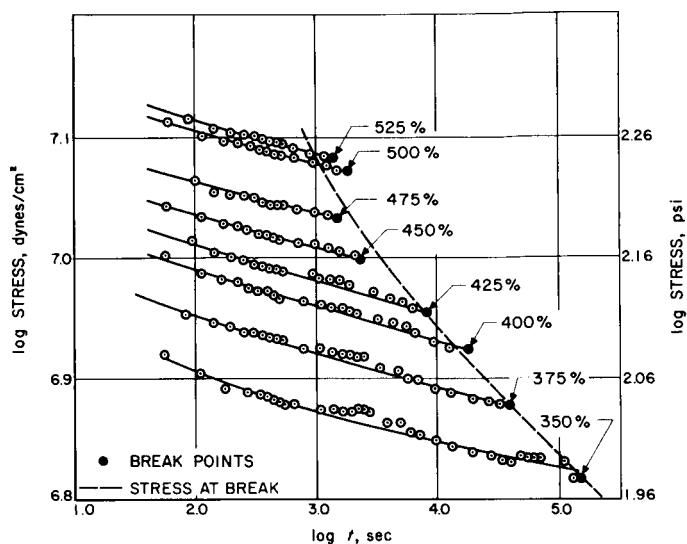

Figure 3. Stress relaxation of an SBR rubber at 1.7°C under elongations from 350 to 525%
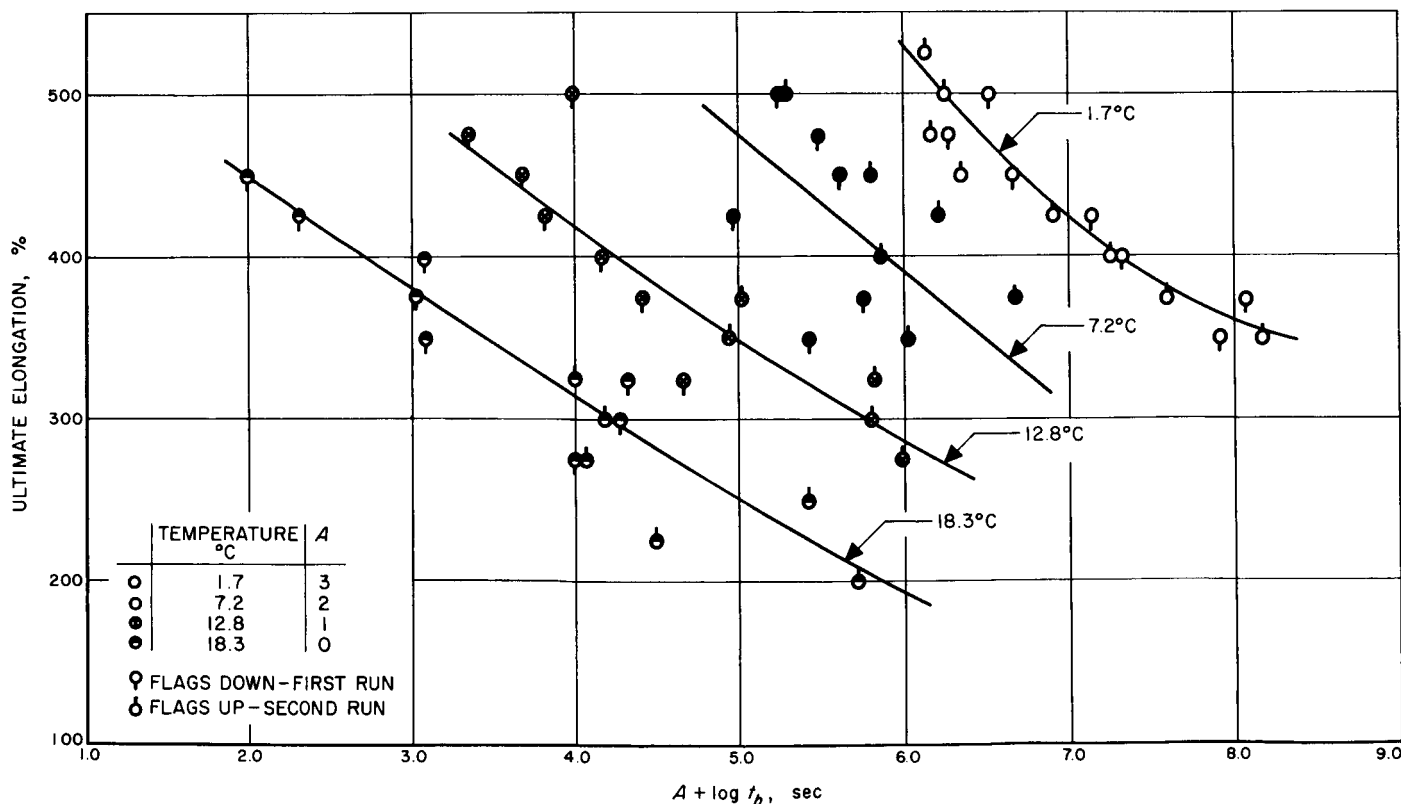
Solid points indicate rupture

Figure 4. Variation of ultimate elongation with log t_b under constant elongations
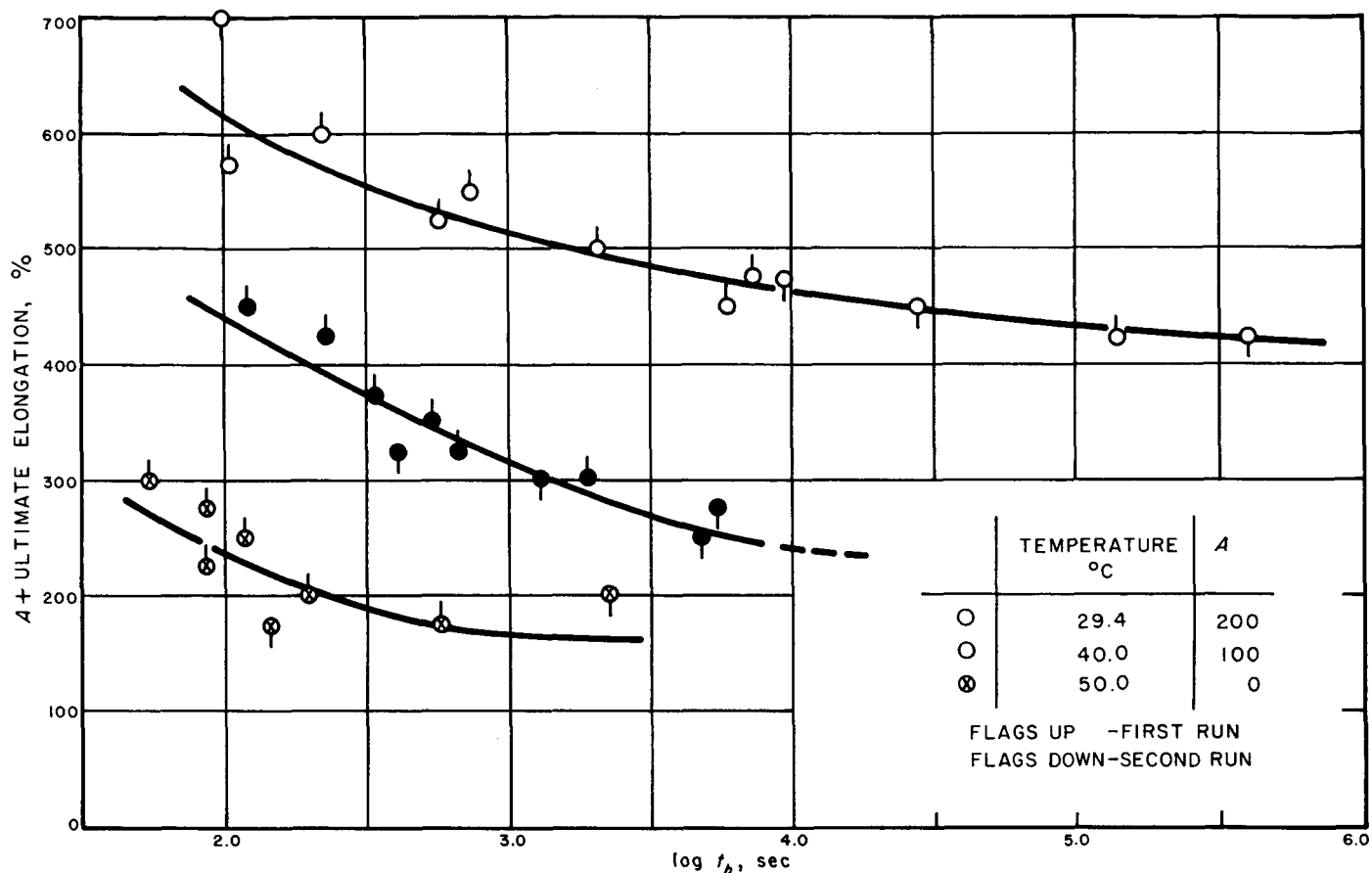
(values of A are arbitrary)

Figure 5. Variation of ultimate elongation with log t_b under constant elongations

(values of A are arbitrary)

only one set of stress measurements were made at 1.7°C, although as shown in Fig. 4, two sets of time-to-break measurements were made at this temperature. (For clarity, the data in Figs. 4 and 6 are shifted along the abscissa by arbitrary amounts A and, similarly, along the ordinate in Figs. 5 and 7.) Because elastic retractive forces are directly proportional to the absolute temperature, all values of the stress-at-break are multiplied by $286/T$ to reduce them to 286°K. (The ultimate property data obtained at 60°C under the constant strain conditions are shown later by the points in Figs. 10 and 11.) Because values of the ultimate properties are by nature rather nonreproducible, the scatter of the points about the lines shown in the Figures is not unduly great, except for the data at 7.2°C.

Both the ultimate elongation and stress-at-break data at the various temperatures were superposed by shifting along the log t_b axis to obtain values of the shift factor a_T. The shape of some curves representing data at the higher temperatures changes markedly with temperature, and the shift-distance required to superpose these

curves was selected somewhat arbitrarily. For the 29.4 and 40°C data, the best over-all superposition—which actually is rather poor—is effected by an unusually large shift. Because this large shift was inconsistent with that required to give superposition of other data separated by 10°C, superposition was effected by considering only the first three or four points at the low end of the time-scale at 40°C. This procedure gave a reasonably realistic value of the shift factor. In general, however, the accuracy of the a_T values is markedly lower than of those presented previously (Ref. 1), obtained by superposing ultimate property data measured at different strain rates. However, values obtained for log a_T are compared in Fig. 8 with the WLF equation. The solid square point in Fig. 8 shows that 12.8°C was selected as the reference temperature for the experimental determination of a_T values by shifting data.

Figure 8 shows that at 29.4°C and higher temperatures the values of log a_T obtained from the stress-at-break data are not in close agreement with those from the ultimate elongation data. Actually, values of log a_T
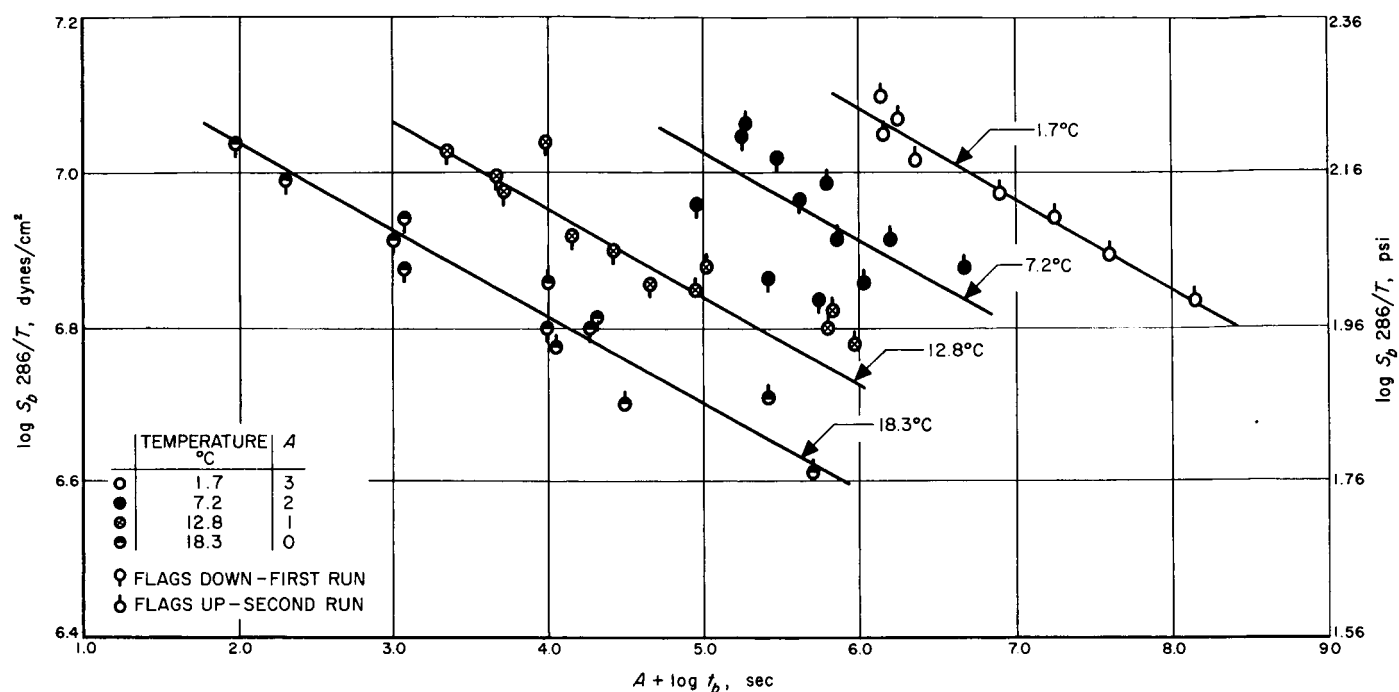
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Figure 6. Logarithm of the stress-at-break reduced to 286°K (S_b 286/T) plotted against log t_b

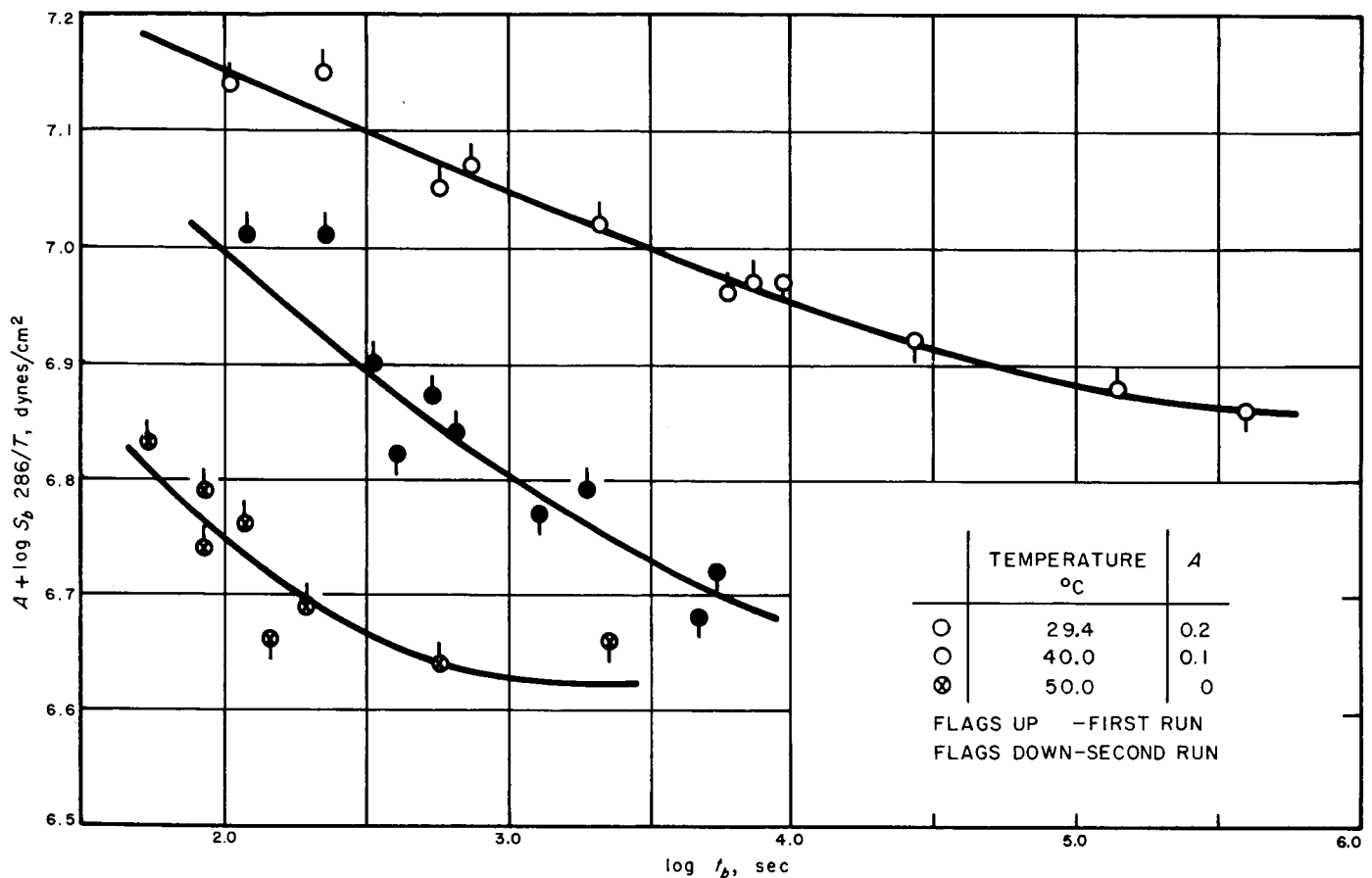Data obtained under constant elongations (values of A are arbitrary)

from the stress-at-break data seem to be in moderate agreement with the WLF equation except at 50 and 60°C, whereas values from the elongation data begin to depart markedly from the WLF equation at temperatures above 29.4°C. A small deviation from the WLF equation might be expected at temperatures above about 40° because this equation often applies only at temperatures less than $T_s + 50$; for the SBR, $T_s + 50$ equals 40°C.

Because of the poor accuracy of the a_T values, the only conclusion that can be drawn is that they are not grossly inconsistent with the WLF equation. However, the data certainly do not prove that rupture under conditions of constant strain is controlled entirely by the internal viscosity as would be shown if the values agreed closely with the WLF equation. Actually, the stress relaxation data, discussed elsewhere (Ref. 7), suggest that some chemical degradation occurred during the experiments at 50 and 60°C. Although the thermostat was flushed continuously with dry nitrogen, as mentioned under Sec. II-B, the oxygen concentration in the thermostat was only one-third that in air. Thus, the nitrogen purge was ineffective to prevent possible degradation. If degradation occurred, the values of log a_T obtained by superposing ultimate property data would be expected to differ from those predicted by the WLF equation. However, the stress relaxation data

showed degradation only after about 6×10^4 sec at 50 and 60°C; under these conditions, only two values of the ultimate properties were obtained (see Figs. 10 and 11). Thus, there is no definite evidence that rupture times were influenced by degradation, although it is conceivable that ultimate property data are affected to a greater extent by small amounts of degradation than are stress relaxation data.

Because of the difficulty in choosing a reliable set of log a_T values, the ultimate properties at the various temperatures are not shown plotted against the time-to-break reduced to a common temperature. Although such reduced plots can be constructed, their reliability for predicting values of the ultimate properties over an extended time-scale at different temperatures would be questionable. Values of the ultimate properties observed under constant strain conditions at a given temperature will thus be compared directly with the values measured at different strain rates.

The ultimate properties at different strain rates were measured at seven temperatures between −5 and 80°C and at three strain rates at most of these temperatures. The strain rates ranged from 1.52×10^{-4} to 1.52×10^{-1} sec^{-1}, although this range was not covered at any single temperature. The few data obtained at each temperature coupled with the poor reliability of individual

Figure 7. Log S_b 286/T plotted against log t_b

Data obtained under constant elongations (values of A are arbitrary)

values of the ultimate properties precluded the deter-
mination of accurate values of log a_T by shifting plots
of the data at each temperature to effect superposition.
Because the previous study (Ref. 1) showed that values
of a_T given by the WLF equation can be used to effect
superposition, the present data were superposed by
using the a_T values shown by the plot of the WLF equa-
tion in Fig. 8. The resulting curves are shown in Fig. 9
where the ultimate strain and log S_b 286/T are plotted
against log t_b/a_T, the quantity t_b/a_T being the time-to-
break reduced to 286°K.

To compare the data measured at constant strain rates
with those at constant strains, the curves in Fig. 9 were
reduced to each of the temperatures at which constant
strain measurements were made; this reduction was
made by using values of log a_T given by the WLF equa-
tion in Fig. 8 to change the time-scale. Sections of the
curves reduced to 29.4, 40, 50, and 60°C are compared
in Figs. 10 and 11 with the constant strain data meas-
ured at these temperatures. In the Figures, sections of
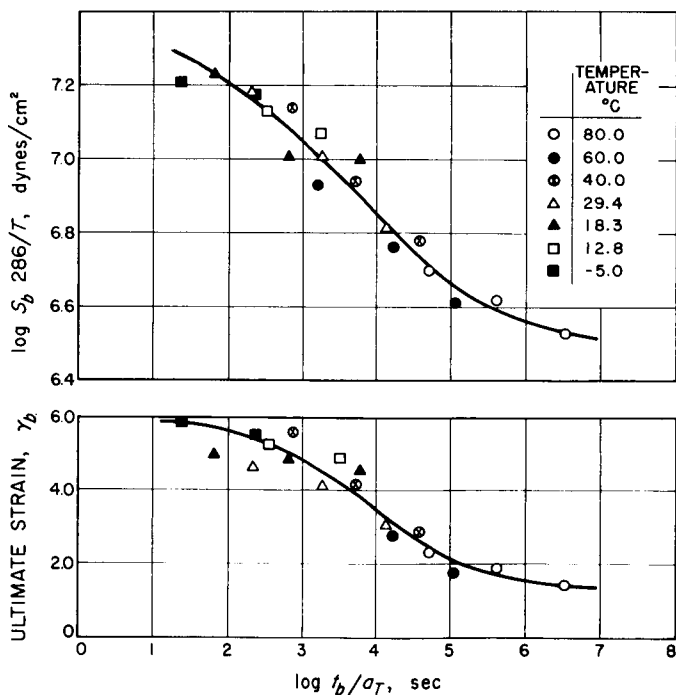the reduced curves are shown by solid lines and the

individual values measured at constant strain rates at
29.4, 40, and 60°C (measurements were not made at
50°) are shown by points; the dotted lines show the
constant strain data, and individual values obtained
under these conditions are shown at 60°C. Plots like
those in Figs. 10 and 11 were also prepared from data
at 1.7, 7.2, 12.8, and 18.3°C. Because these plots did not
show any trends not shown by Figs. 10 and 11, they are
not presented.

Figures 10 and 11 show that most individual values
obtained at constant strain rates lie to the right of the
curves showing values measured at constant strain.
Although the curves showing the constant strain rate
data lie to the right of those showing constant strain
data—at least at shorter times—the curves at 29.4°C
intersect so that at long times the constant strain rate
data lie to the left of the constant strain data. This cross-
over is unexpected and probably is not real, especially
since no individual values measured at constant strain
rate lie below or to the left of the curves showing the
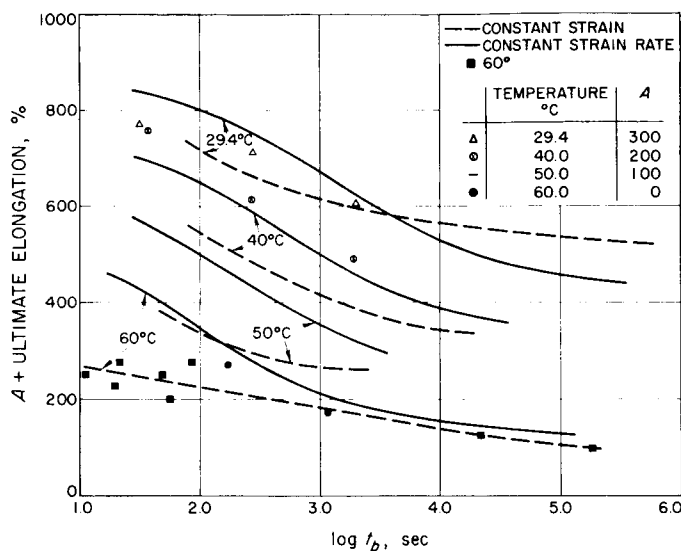constant strain data.
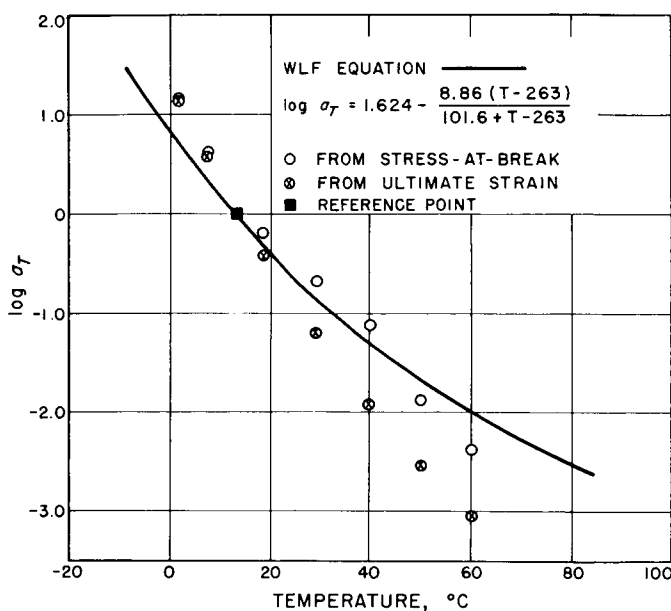
Figure 8. Shift factor log a_T obtained by superposing ultimate property data compared with the WLF equation

The factor 1.624 allows for the fact that the reference temperature here is 12.8°C instead of −10°C

A crossover of the curves indicates that at long times the time-to-break for a sample under constant strain is greater than for a sample subjected to a constant strain rate. However, under constant strain conditions, the energy input to a sample as measured by the product of stress and elongation is considerably greater than the

Figure 10. Comparison of ultimate elongation vs log t_b data measured at constant elongations with values measured at different strain rates

(values of A are arbitrary)

Figure 9. Ultimate properties measured at different strain rates and temperatures plotted against log t_b

Data reduced to 13°C
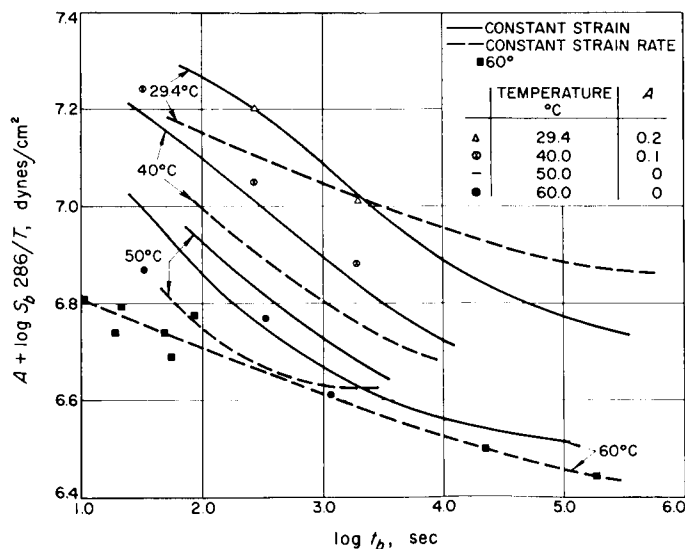
Figure 11. Comparison of log S_b 286/T vs log t_b data measured at constant elongations with values measured at different strain rates

(values of A are arbitrary)

energy input to a sample which is strained continuously at a constant rate until rupture occurs. This factor suggests that the time-to-break for samples under constant strain should not be longer than for samples subjected to a constant strain rate. Thus, the crossover is tentatively attributed to some error in either the experimental data or its reduction.

An attempt was made to measure ultimate properties under constant elongations at -5 and $-15°C$. The results were erratic, and thus are not presented. At these low temperatures, it is possible that during the stretching of a specimen to certain elongations, the left side of the bell-shaped curve—like that shown in Fig. 1—was often approached rather closely. If this were true, the time of stretching a specimen could affect the results because a small difference in time would change markedly the closeness of approach to the bell-shaped curve. Thus, in some tests damage may have been done to the network when a specimen was stretched; this damage could cause the specimen to break almost immediately or to break at times considerably different than expected; both types of behavior were observed. When the bell-shaped curve is not closely approached during the stretching of a specimen, no damage should be done to the specimen. However, the time-to-break might now be greater than the 4 to 5 days normally used for a test, and no rupture of the specimen would be observed.

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